DECLARATION

Docket No.: 21107/0207506-US0
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Jae Kap Lee

Application No.: 10/574,264 — Confirmation No.: 7194

Filed: March 31, 2006 — Art Unit: 1792

For: DIAMOND SHELL WITH A GEOMETRICAL FIGURE AND METHOD FOR FABRICATION THEREOF — Examiner: J. A. Miller

DECLARATION OF JAE KAP LEE AND PHILLIP JOHN UNDER 37 C.F.R. § 1.131

MS Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

I, Jae Kap LEE and I, Phillip JOHN, do hereby declare and state as follows:

1. We are inventors of the subject matter described and claimed in the above-identified patent application.

2. Prior to the filing date of the Kley patent (U.S. Patent No. 7,309,446), we had conceived our invention as described and claimed in the subject application in the United Kingdom, a WTO member nation since January 1995.

3 As evidence that our work antedates the Kley patent, we refer to the invention disclosure attached hereto as Exhibit 1. Dates, along with privileged information, appearing in this document have been redacted. Exhibit 1 discloses, in text and drawings, conception of the invention of the present application from a time prior to the filing date of the Kley patent.

4 The conception of the invention was coupled with diligence in reduction to practice from a time prior to the filing date of the Kley patent and ending with the constructive reduction to practice of the invention — i.e., the filing of the present application's priority document (KR Application No. KR 10-2004-0083710, filed October 19, 2004).

5. As evidence of due diligence, we refer to another "Innovation Disclosure Form (IDF)" to revise and update IDF as Exhibit 1, attached hereto as Exhibit 2. Also, we refer to an overview and schematic of the patent revised on 28 May 2004, attached hereto as Exhibit 3. Exhibits 2~3 show progression of the experiments. Final revision date of Exhibits 2~3 is shown by screen-print attached hereto as Exhibit 4. Also, we refer to pages of the disclosure of the subject application that I personally prepared, attached hereto as Exhibit 5. We refer to a translation of the disclosure of Exhibit 5, attached hereto as Exhibit 6. We refer to a document that was used to transmit the disclosure of Exhibit 5 to the proper personnel to be used for filing a patent application for the invention, attached hereto as Exhibit 7. A copy of the translation of the Exhibit 7 is also provided as Exhibit 8. The transmission of Exhibit 5 is dated July 8, 2004. We refer to the assignment of the disclosure of Exhibit 5 to the assignee of the subject application, attached hereto as Exhibit 9. A copy of the translation of the assignment is also provided as Exhibit 10.

6. We further declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: 10 March 2009

Jae-Kap Lee

Jae Kap Lee, Inventor

Dated: 10 March 2009

Phillip John

Phillip John, Inventor

EXHIBIT 1

CONFIDENTIAL WHEN COMPLETED

Technology & Research Services
Heriot-Watt University

INNOVATION DISCLOSURE FORM (IDF)

Dear Innovator(s)

Thank you for completing this form and providing as much information as you can about your innovation.

Technology & Research Services (TRS) will undertake an initial investigation of the commercial potential of your innovation and a prior art search. If this is encouraging, TRS, with your help, will produce a Preliminary Business Case and present the opportunity to the next Innovation Exploitation Board (IEB) meeting.

If the IEB support the case then TRS resources will be utilised to fully investigate the commercial exploitation of your innovation and file a patent application if appropriate. Therefore it is important you provide us with as much information as possible about your invention. Please note that all boxes are expandable to accommodate as much information as you have.

Please return the completed IDF along with any supporting documents by e-mail or internal post to:

Mike Cox, Technology Transfer Manager, Technology & Research Services

m.k.cox@hw.ac.uk

For TRS Use Only:

Received on:_____ Case Number:_____

1. Summary of Your Innovation

1.1 Title of your innovation

A method for the fabrication of micro-diamond hollow shells

1.2 Single sentence description of the unique aspect(s) of your innovation

A controlled method for producing inert hollow diamond shapes for storage, preservation and transporting of materials with controlled release properties.

1.3 Field of science/technology your innovation belongs to

Diamond coating technology

1.4 On what date did you first make notes or sketches about your innovation? Is this in Laboratory Notebook(s), computer disc, etc?

2. Technical Description of Your Innovation

2.1 In a few sentences please explain the particular problem that your innovation solves?

This technique provides a technique for the fabrication of hollow three dimensional shapes of CVD diamond.

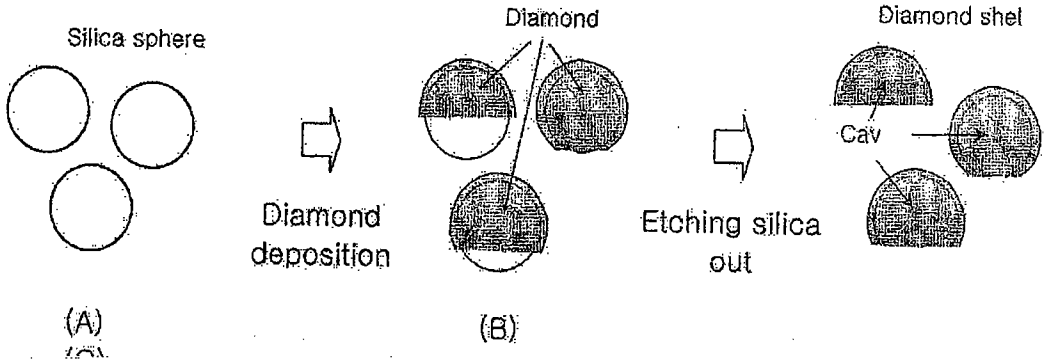
Current methodologies that the team are aware of can only produce shapes at the macro scale but this technology provides a process for producing microscale hollow shapes.

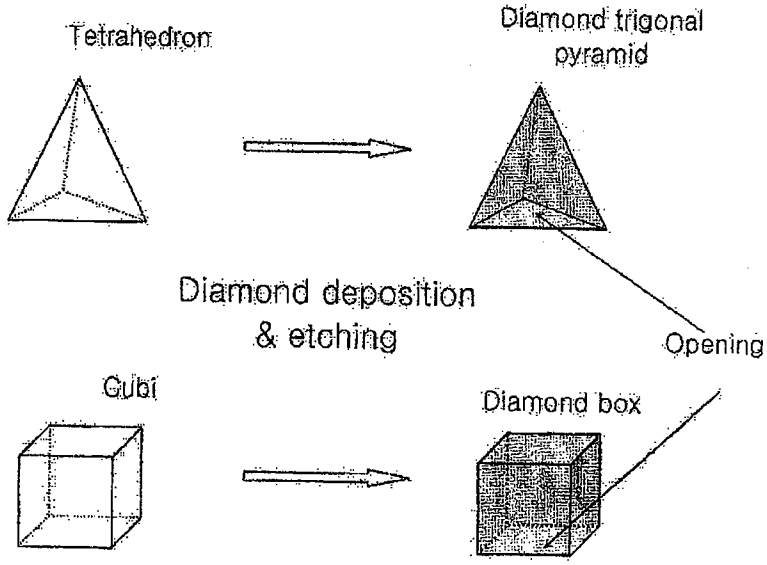
2.2 Background to your innovation: please describe the closest prior public knowledge (prior art) system(s)/method(s) you are aware of, and any disadvantages they possess.

2.3 What is new or unique about your innovation? How does it improve on the closest prior art described above?

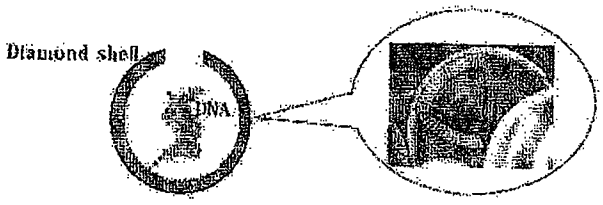
This technique provides micron scale hollow shapes. The hollow shapes have an aperture which allows material to be placed inside the shape for storage, preservation or transport purposes. The technology will allow the size of the aperture to be controlled providing a method for controlling the rate of release of material from the hollow shell.

2.4 Please explain your invention fully, so that someone with reasonable technical knowledge could reproduce it. (Please attach any diagrams, sketches, equations, etc which may be useful or relevant).





The technique involves coating the 3D surface object with CVD diamond then chemically etching out the support material leaving a hollow diamond shell. Any shape can be coated.



The hollow diamond shell can then be filled with biological or chemical materials. Enhanced attachement can be achieved by using a variety of methods to the diamond

surface e.g. covalent linkages.

2.5 Please give one or more particular examples of how your invention may be performed in practice, including a description of the preferred or ideal example. (Please include experimental results and results of trials on prototypes if available.).

2.6 Please summarise the technical features of your innovation that are essential to its working and those that are important but not essential.

- Nucleation conditions
- Separation of support from nucleation material
- Conditions to provide a continuous CVD film over support surface
- Leave an area uncovered by diamond coating to allow etching material to enter to remove support. This is done either by controlling process or including a blocking agent
- Conditions to prevent micro-supports from coalescing

2.7 What do you see as the main inventive concept of your innovation?

The inventive concept is the bringing together of a number of standard processes to provide hollow shapes for transport, preservation and storage of material. Standard processes for

2.8 What do you see as the main technical advantages and benefits of your innovation? Please make a distinction between commercial advantages and technical advantages.

The technique provides a method for the production of inert microscopic diamond shells which can be used for the preservation, storage and transport and delivery of molecules in a controlled fashion.

An inert material such as diamond provides advantages in that there is no cross reactivity of the carrier shell with either the contents of the shell or the medium into which is being introduced.

The ability to control the aperture of the shell opening is attractive in that the leakage of the molecule from the shell can be controlled.

Ongoing research in the group is looking at the attachment mechanisms for molecules to the diamond surface which would provide further control on the release or retention of molecules within the shell.

2.9 Do you see your invention as a "basic concept" invention, or more as an "improvement"?

2.10 What stage of development is your innovation at (e.g. concept, prototype, computer simulation, etc)? If you have built a prototype and/or carried our experiments, have you achieved successful results? Please describe your plan to achieve a prototype if not at that stage.

2.10 Thinking laterally – could your invention have applications in technical fields other than your own field of research? If so please explain.

3. Ownership

3.1 Has anyone other than yourself ever worked on this innovation (e.g. student, research fellow, company employee, visiting fellow, consultant, etc)? If "Yes" please give details.

3.2 Please give the FULL name, including middle names, complete home address (not University address), including postcodes, citizenship, University Department (if possible) and status within the University of **each** individual involved in this innovation. Please indicate the relative percentage contributions of those having an inventive (non-routine) input.

3.3 Did all the inventors create the innovation entirely while **employed** by Heriot-Watt University? If not please give details.

3.4 Was the work on this innovation funded in any way from any source outside Heriot-Watt University (e.g. research council, government grant, European funding, industrial sponsorship, consultancy, etc)? Please give details of all funding used on this innovation, including research grant number(s).

3.5 Did this funding involve a signed Agreement (e.g. intellectual property or consortium agreement)? If "Yes" please give details and include a copy of the agreement.

4. Disclosure / Publication History

4.1 Have any details of, or information on, your innovation been

- Published, or submitted for publication, in a journal, book, research memorandum, abstract, conference proceedings, trade press, newspapers, magazines, publicity handout, University prospectus or annual report, etc
- Displayed on a website or in an electronic journal
- Included in a poster display within the University, at a conference, etc
- Included in a research or grant application
- Included in a student's thesis, paper, poster, etc
- Disclosed in a non-secret/non-confidential manner in any other written or oral form

If "Yes" please give specific details including the date(s) of disclosure.

4.2 Have you (or anyone else involved with your innovation) ever communicated about your innovation to anyone not employed by Heriot-Watt University, including students, (including funding applications and reports, conversations, telephone, meetings, letters, e-mail, presentations at conferences, television, radio, etc.)? Have you (or any one else involved with your innovation) ever demonstrated a prototype to anyone not employed by Heriot-Watt University, including students? If "Yes" please give details.

4.3 Is there any planned date for a first (non-confidential) disclosure of the invention (e.g. publication, conference presentation or poster, etc)? If yes please give the date and details of the planned disclosure.

4.4 Please give details of any previous patent applications by the inventor(s) in this field, including any for the present invention, and including any discontinued patent applications.

5. Prior Art (Publicly available knowledge)

5.1 Please summarise all prior art (disclosed or published work in this area) that you are aware of or have found (indicating the main sources of information).

5.2 To facilitate searching for prior art please supply:

Names of leading researchers in this field

Names of companies working in this field

Key descriptive words to search on

6. Commercial Opportunity

6.1 What are the applications or fields of use of your innovation?

6.2 Please list the unique benefits of your innovation to a customer/user

6.3 Why would companies use your innovation rather than the current techniques/solutions/approaches?

6.4 What potential blocks are you aware of that might prevent companies using your innovation (e.g. switching costs, entrenched competition, testing requirements, legislative restrictions etc)?

6.5 Give the names of any companies that you have identified who would be interested in your innovation.

6.6 What geographical territories do you think would be the main markets for your invention?

6.7 Please summarise any market volume or value information you have and the source(s). In particular, please estimate the value of this innovation.

6.8 Please summarise how you think the commercial opportunity you have identified might be exploited, including route to market.

6.9 Please estimate the time required for your innovation to be ready for exploitation (i.e. when could you have a product/service ready to be offered for sale) and the cost required to reach this point?

Declaration

I certify that, to the best of my abilities, the information given on pages 2-10 is true and accurate.

Professor Phillip John
Signed

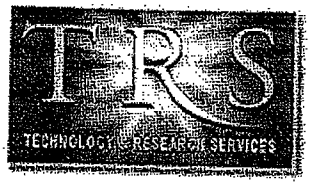
Date

Signed
Dr Jae-Kap Lee

Date

EXHIBIT 2

CONFIDENTIAL WHEN COMPLETED



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Heriot-Watt University

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Mike Cox, Technology Transfer Manager, Technology & Research Services

m.k.cox@hw.ac.uk

For TRS Use Only:

Received on:_____ Case Number:_____

1. Summary of Your Innovation

1.1 Title of your innovation

A method of fabrication of diamond shells

1.2 Single sentence description of the unique aspect(s) of your innovation

This innovation provides a method for fabrication of diamond shells of which size ranges from several um to mm.

1.3 Field of science/technology your innovation belongs to

Material

1.4 On what date did you first make notes or sketches about your innovation? Is this in Laboratory Notebook(s), computer disc, etc?

2. Technical Description of Your Innovation

2.1 In a few sentences please explain the particular problem that your innovation solves?

This innovation enables us to make diamond shells, of which sizes range between several um and few mm, which have never been fabricated yet.

2.2 Background to your innovation: please describe the closest prior public knowledge (prior art) system(s)/method(s) you are aware of, and any disadvantages they possess.

Diamond has eminent chemical and physical properties. Diamond has been synthesized by high pressure and high temperature (HPHT) method since 1955 and chemical vapour deposition (CVD) method since 1980s. The typical shape of HPHT diamond is powder (um), grit(hundreds um) or bulk(few mm), while that of CVD diamond is film which grows on a substrate.

The appearance of the CVD diamond films is determined by the shape of the substrate used, whether they are subjected to or free from the substrate. The dimension of the diamond films is up to several cm^2 of which the thickness can be controlled by deposition time from nm to mm. Freestanding diamond films can be made by removing the substrate after deposition.

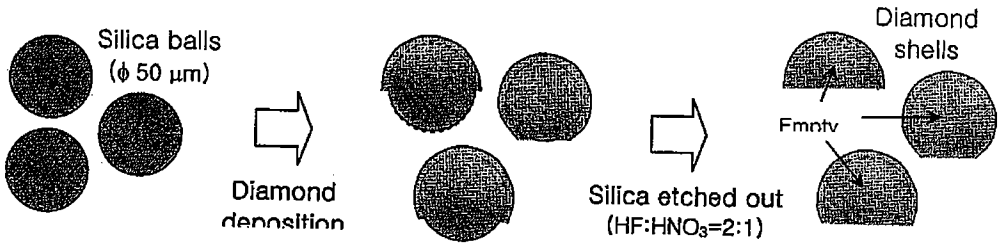
Conventional shapes of diamond are powder, grit, bulk or film. No shell-typed diamonds hollow are reported.

2.3 What is new or unique about your innovation? How does it improve on the closest prior art described above?

This innovation provides a method to fabricate diamond shells in a range of several um to mm in dimension, of which inside is vacant. Figures (external shape) of the diamond shell can be changeable-spherical, triangular or rectangular etc..

2.4 Please explain your invention fully, so that someone with reasonable technical knowledge could reproduce it. (Please attach any diagrams, sketches, equations, etc which may be useful or relevant).

Diamond films are deposited on a spherical silica substrate by conventional CVD method, but not covers its surface fully. The silica ball served as a substrate is etched out in an acid solution. As a result, diamond shells can be fabricated. The appearance of the diamond shell is changeable by using different shape of the substrate. Procedure is shown as follow;



2.5 Please give one or more particular examples of how your invention may be performed in practice, including a description of the preferred or ideal example. (Please include experimental results and results of trials on prototypes if available.)

The diamond shell can be used as a new container for a valuable and feeble miniature. An example is a DNA preservatory.

2.6 Please summarise the technical features of your innovation that are essential to its working and those that are important but not essential.

This innovation is a marvellous combination of two well-known techniques, CVD diamond deposition and etching the silica substrate out.

2.7 What do you see as the main inventive concept of your innovation?

Etching the silica substrate served for diamond coating out

2.8 What do you see as the main technical advantages and benefits of your innovation? Please make a distinction between commercial advantages and technical advantages.

Fabrication of diamond with a new shape of miniature shell by using conventional well-known techniques. These diamond shells can provide a new application commercially available.

2.9 Do you see your invention as a "basic concept" invention, or more as an "improvement"?

2.10 What stage of development is your innovation at (e.g. concept, prototype, computer simulation, etc)? If you have built a prototype and/or carried our experiments, have you achieved successful results? Please describe your plan to achieve a prototype if not at that stage.

2.10 Thinking laterally – could your invention have applications in technical fields other than your own field of research? If so please explain.

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3.3 Did all the inventors create the innovation entirely while **employed** by Heriot-Watt University? If not please give details.

3.4 Was the work on this innovation funded in any way from any source outside Heriot-Watt University (e.g. research council, government grant, European funding, industrial sponsorship, consultancy, etc)? Please give details of all funding used on this innovation, including research grant number(s).

3.5 Did this funding involve a signed Agreement (e.g. intellectual property or consortium agreement)? If "Yes" please give details and include a copy of the agreement.

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4. Disclosure / Publication History

4.1 Have any details of , or information on, your innovation been

- Published, or submitted for publication, in a journal, book, research memorandum, abstract, conference proceedings, trade press, newspapers, magazines, publicity handout, University prospectus or annual report, etc
- Displayed on a website or in an electronic journal
- Included in a poster display within the University, at a conference, etc
- Included in a research or grant application
- Included in a student's thesis, paper, poster, etc
- Disclosed in a non-secret/non-confidential manner in any other written or oral form

If "Yes" please give specific details including the date(s) of disclosure.

No

4.2 Have you (or anyone else involved with your innovation) ever communicated about your innovation to anyone not employed by Heriot-Watt University, including students, (including funding applications and reports, conversations,

telephone, meetings, letters, e-mail, presentations at conferences, television, radio, etc.)? Have you (or any one else involved with your innovation) ever demonstrated a prototype to anyone not employed by Heriot-Watt University, including students? If "Yes" please give details.

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No

4.4 Please give details of any previous patent applications by the inventor(s) in this field, including any for the present invention, and including any discontinued patent applications.

No

5. Prior Art (Publicly available knowledge)

5.1 Please summarise all prior art (disclosed or published work in this area) that you are aware of or have found (indicating the main sources of information).

5.2 To facilitate searching for prior art please supply:

Names of leading researchers in this field

Names of companies working in this field

Key descriptive words to search on

6. Commercial Opportunity

6.1 What are the applications or fields of use of your innovation?

DNA preservation or DNA microarray

Miniature container

6.2 Please list the unique benefits of your innovation to a customer/user

New shape of diamond

Absolutely stable miniature container

6.3 Why would companies use your innovation rather than the current techniques/solutions/approaches?

There was no this kind of diamond

6.4 What potential blocks are you aware of that might prevent companies using your innovation (e.g. switching costs, entrenched competition, testing requirements, legislative restrictions etc)?

6.5 Give the names of any companies that you have identified who would be interested in your innovation.

De beers

6.6 What geographical territories do you think would be the main markets for your invention?

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Declaration

I certify that, to the best of my abilities, the information given on pages 2-10 is true and accurate.

---------- ----------

Signed Date

EXHIBIT 3

Joint patent application
(to be submitted to Korea)

14/5/2004

- **Title: A method for fabrication of micro diamond shells**
- **Inventors:** Jae-Kap Lee and Phillip John (Contribution: each 50%)
- **Applicants:** KIST and Heriot-Watt university

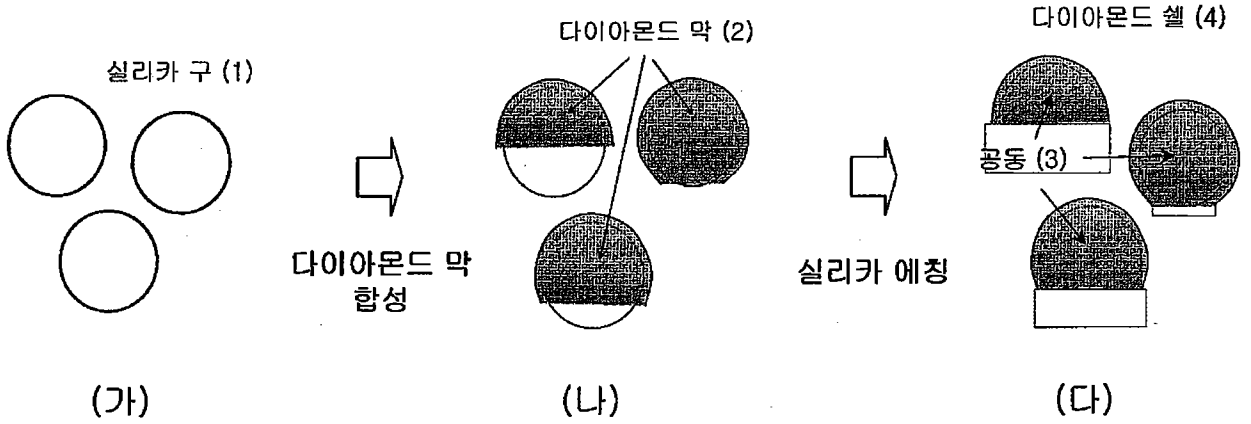
  -The expenses for the joint patent application is borne 50% by KIST and 50% by Heriot-Watt university.

- **Claims,**

  1. In conventional chemical vapor deposition(CVD) diamond synthesis, a method fabricating diamond shells, of which the inside is vacant, by etching a spherical substrate out after a self-standing diamond film is coated on its surfaces, but not covered fully.
  2. The method of claim 1, wherein the spherical substrate is between 1-1000 um in diameter.
  3. The method of claim 1, wherein the substrate can have other geometries, such as tetrahedron and cube.

• **Figures**

Figure 1



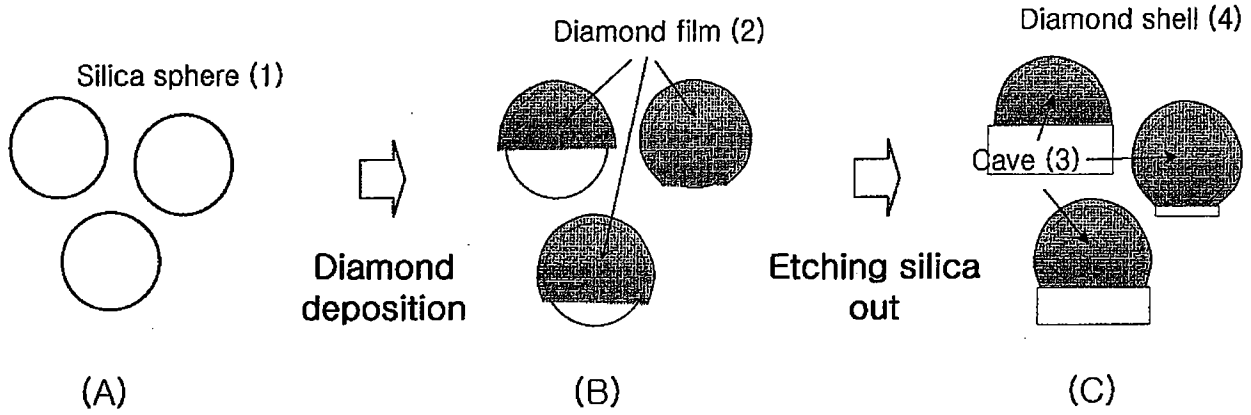
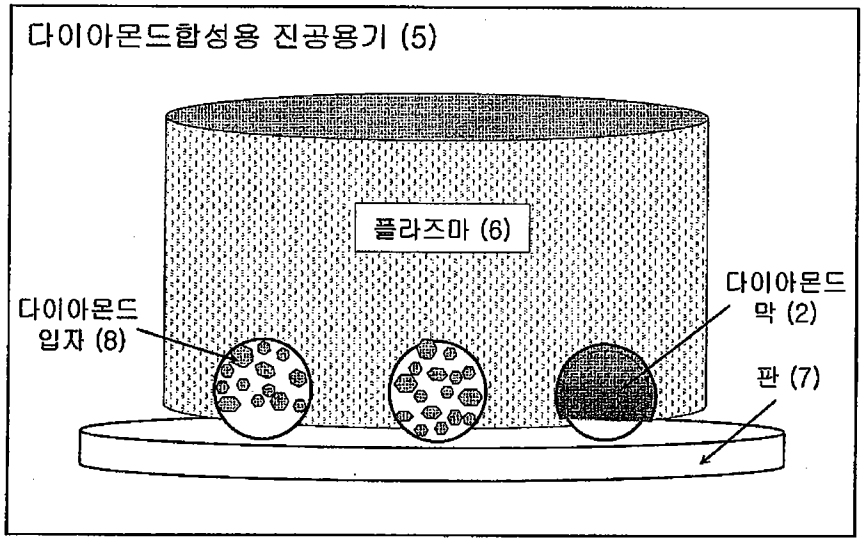
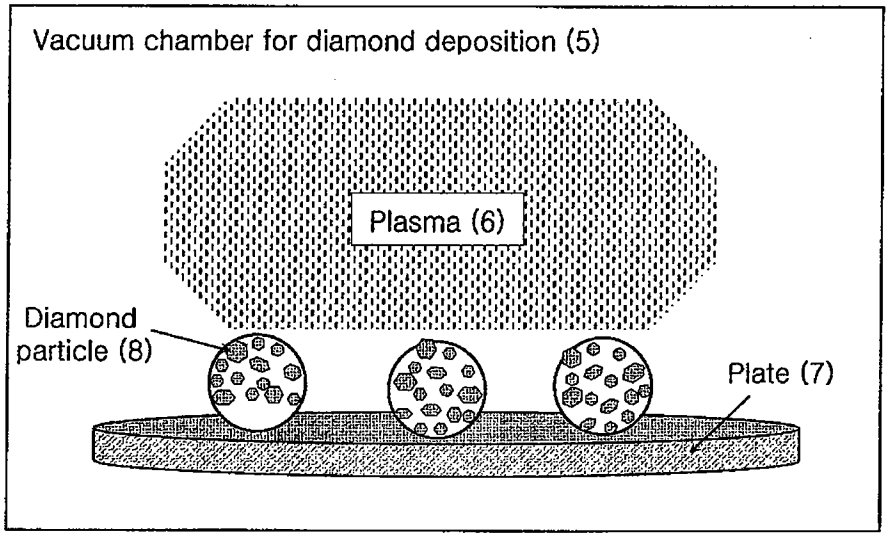
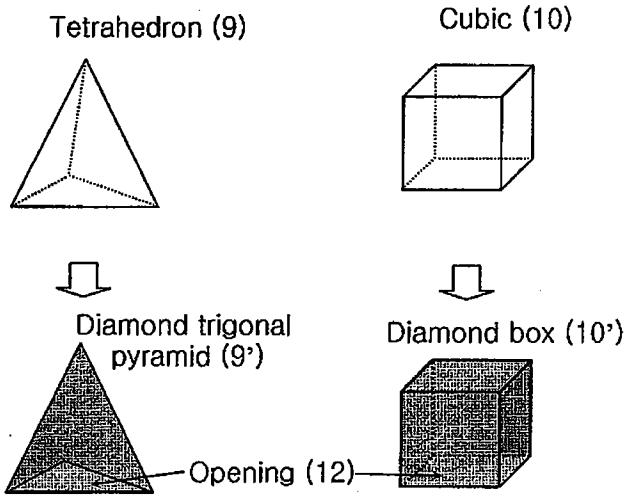


Figure 2


다이아몬드합성용 진공용기 (5)
플라즈마 (6)
다이아몬드
입자 (8)
다이아몬드
막 (2)
판 (7)

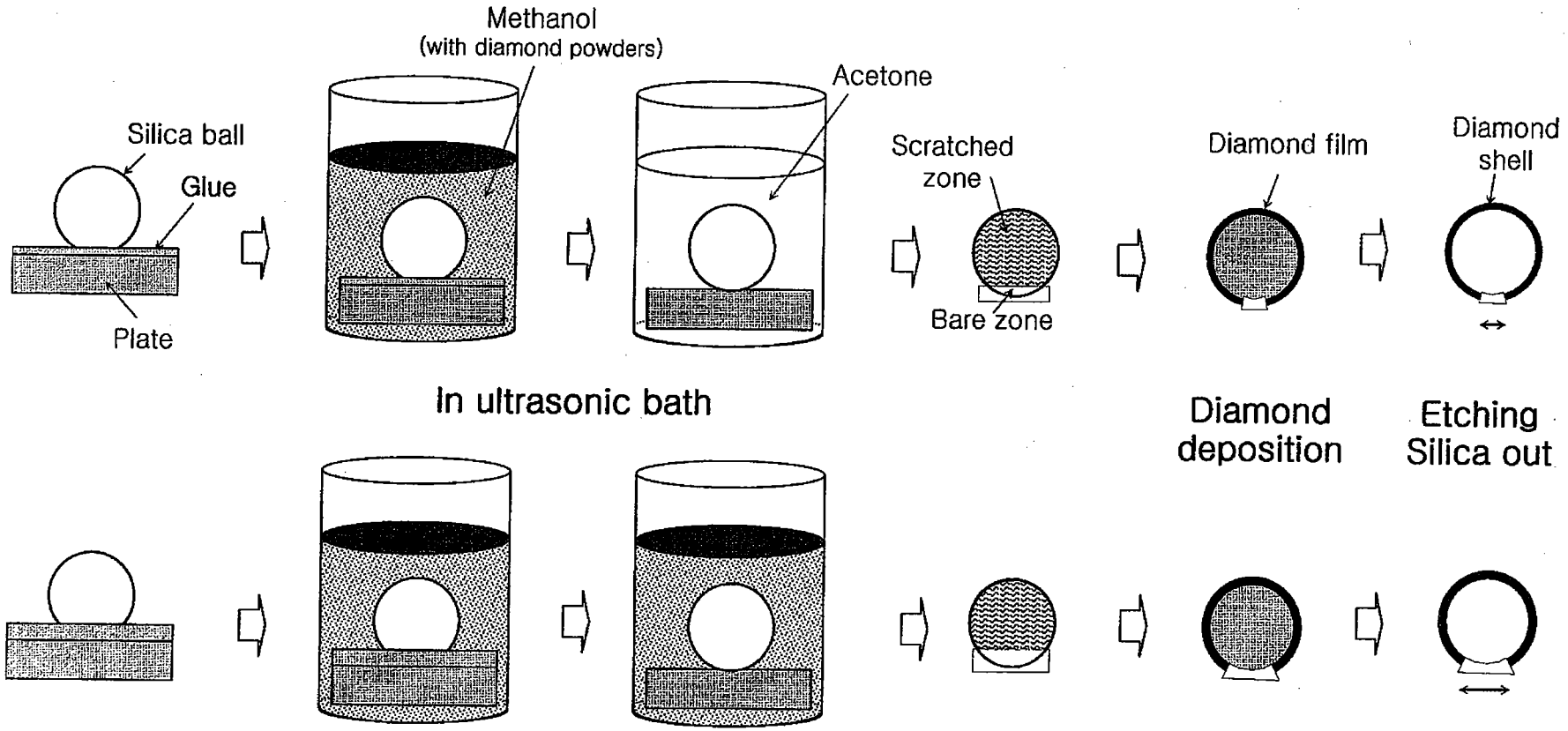

Vacuum chamber for diamond deposition (5)
Plasma (6)
Diamond
particle (8)
Plate (7)

Future works


Tetrahedron (9)
Cubic (10)
Diamond trigonal pyramid (9')
Diamond box (10')
Opening (12)

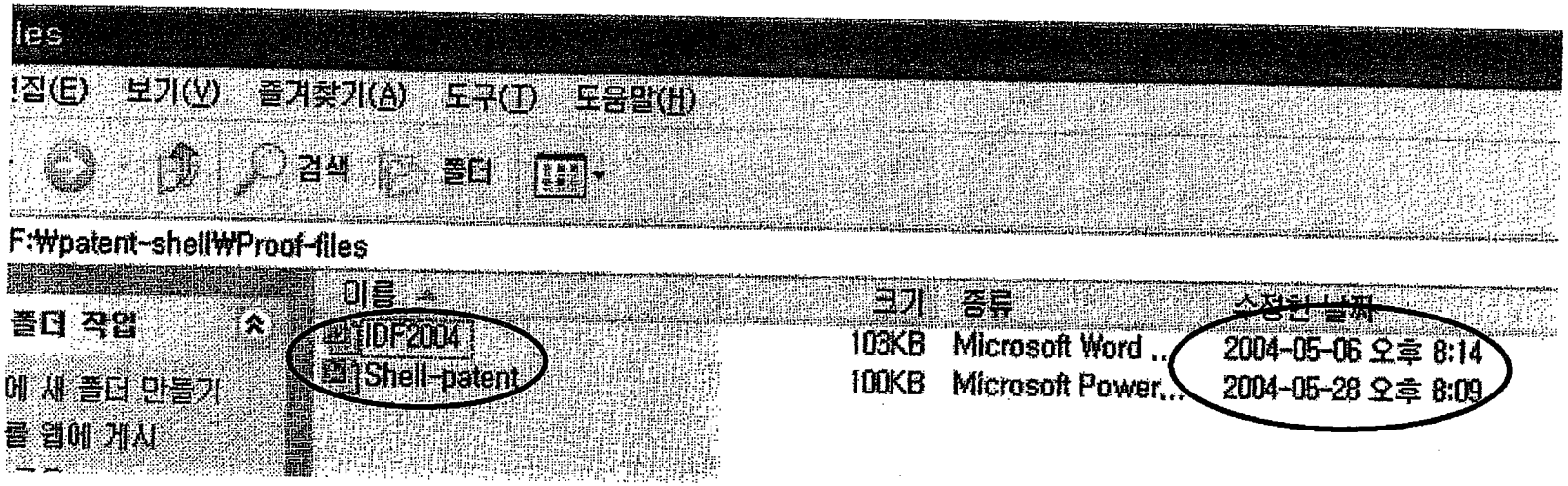
(가) (나)

- **Control of the size of shell's opening**



Glue: unsolvable in Methanol, but solvable in Acetone or...

EXHIBIT 4



Explanation

File name	Date	contents
(Exhibit 2) IDF2004.doc	2004-05-06	IDF, revised on 06 May 2004 (The date, 19/12/2003, appears)
(Exhibit 3) Shell-patent.ppt	2004-05-28	Overview and schematic of the patent, revised on 28 May 2004

EXHIBIT 5

【요약서】

【요약】

본 발명은 마이크로(micro) 크기의 다이아몬드 쉘(diamond shell)을 제조할 수 있는 방법에 관한 것이다. 마이크로 크기를 갖는 구형의 실리카(모재)에, 모재표면의 일부분을 제외하고, 기상화학증착법(CVD)법으로 다이아몬드 막을 증착한 후 모재를 에칭 시켜 제거함으로써, 내부가 비어있는 다이아몬드 구조물(쉘)을 제조할 수 있다. 모재의 모양을 구, 사면체 및 육면체 등으로 변화시키거나, 또한, 모재의 크기를 변화시킴에 의해, 모양 및 크기에 있어서 다양한 다이아몬드 쉘을 제조할 수 있다. 제조할 수 있는 다이아몬드 쉘의 크기는 수 ㎛~수 ㎜이다.

【대표도】

도 1

【색인어】

다이아몬드, CVD, 마이크로, 쉘, 에칭

【명세서】

【발명의 명칭】

다이아몬드 쉘의 제조방법{A method for fabrication of diamond shells}

【도면의 간단한 설명】

도 1은 본 발명의 한 예로, 구상의 실리카(1) 모재 위에, 모재의 일부분을 제외하고, 기상화학증착합성법으로 다이아몬드 막을 증착한 후(나), 실리카를 에칭시켜 제거함으로써, 다이아몬드 쉘을 제조하는 과정을 도식적으로 나타낸 것이다.

도 2는 일반적인 CVD 다이아몬드합성장치를 사용하여 구상의 실리카에 다이아몬드 막을 합성하는 방법을 나타내는 모식도이다.

(도면의 주요부분에 대한 부호 설명 ; 1: 모재(실리카 구), 2: 다이아몬드 막, 3: 공동, 4: 다이아몬드 쉘, 5: 다이아몬드합성 용기, 6: 플라즈마, 7: 판, 8: 다이아몬드 입자

【발명의 상세한 설명】

【발명의 목적】

【발명이 속하는 기술분야 및 그 분야의 종래 기술】

본 발명이 속하는 기술분야는 재료(세라믹)분야이다. 보다 구체적으로, 본 발명은, 세라믹 재료 중의 하나인 다이아몬드를 기상화학증착(CVD)법을 이용하여, 내부가 비어 있는 쉘 모양의 다이아몬드를 제조하는 방법에 관한 것이다.

CVD 다이아몬드합성법(주요 합성조건: 약 100 Torr, 800 ℃)은 1980년대에 개발되어, 지금은 산업에 응용될 수 있는 다양한 CVD 다이아몬드를 생산할 수 있을 정도로 일반화된 기술이다. CVD합성법에서, 다이아몬드는, 진공용기에서 원료 기체(주로 메탄)는 열 또는 플라즈마에 의한 활성화과정에 이어, 모재 위에 입자 형태로 핵생성하고 성장하여 다결정성 막으로 성장한다. CVD법에서 합성법의 종류는 원료기체를 활성화시키는 방법에 따라 분류되는데, 지금까지, hot filament법, 직류전원 플라즈마(Direct current plasma)법, 마이크로웨이브 플라즈마(microwave plasma)법, 직류전원 아크 제트(DC arc jet)법 등 여러 합성법이 개발되어 사용되고 있다. 통상,

판 상의 모재(기판) 위에 수 ㎚~수 백 ㎛ 두께로 증착되는 다이아몬드 막은, 기판에 부착된 상태로 사용되거나(직접코팅: 수 십 ㎛ 이하의 박막), 기판으로부터 분리된 자유막(free standing) 형태(수 백 ㎛ 이상의 후막)로 사용된다. 이와 같이 CVD 합성법에서 얻어지는 다이아몬드의 일반적인 형태는 막상 이다.

CVD법에 앞서, 1955년 미국 GE사에서 개발된 고온고압(high pressure and high temperature(HPHT); 약 5만 기압, 1400 ℃)법에서 합성되는 다이아몬드는 수 백 ㎛ 이하의 크기를 갖는 cubo-octahedron 모양의 입자(grit) 또는 불규칙한 분말(powder)형태이다. 역시 HPHT 조건 하에서 이루어지는 단결정 성장(Single crystal growth)법에서 얻을 수 있는 다이아몬드는 수 mm^3 크기의 bulk 형태이다. 또한, 러시아에서 개발된 폭발(Detonation)법(약 10만 기압~1,000 ℃)에서 얻어지는 다이아몬드는 수 ㎚~수 ㎛ 크기인 모양이 불규칙한 다이아몬드 분말이다. 이와 같이 HPHT법에서 얻을 수 있는 다이아몬드의 일반적인 모양은, 천연다이아몬드처럼, 입자(또는 분말) 형태이다. 이것은 다이아몬드가 탄소의 고압 상이기 때문에, 합성조건이 가혹하고, 이에 따라 얻을 수 있는 다이아몬드의 모양은 제한된다.

다이아몬드를 상압 이하에서 막 상으로 합성하는 CVD법은 다이아몬드의 크기 및 모양의 제약을 많이 극복할 수 있는 계기를 마련하여, 다이아몬드를 수-수 십 ㎠ 크기의 다이아몬드 판을 제작하거나, 직경이 수 ㎠되는 돔(dome) 형태의 다이아몬드 자유 막을 제작할 수 있었다(두께는 약 2 mm 이하).

그러나, 기존 CVD 합성기술로, 크기가 마이크론 단위이며, 내부가 비어있는 쉘 구조를 갖는 다이아몬드의 제조는 불가능하였다.

【발명이 이루고자 하는 기술적 과제】

본 발명은 종래의 기술로는 제조할 수 없는 쉘(shell) 구조의 다이아몬드를 제조하기 위하여 안출된 것으로, 특히, 수 ㎛~수 ㎜ 범위 내에서 원하는 크기의 다이아몬드 쉘을 제조할 수 있는 방법에 관한 것이다. 이 발명은 수 ㎛~수 ㎜ 범위 내에 있는 구형의 모재(실리카)에, 모재 표면의 일부분을 제외하고, 일정 두께의 다이아몬드 막을 증착한 후, 모재를 화학약품을 사용하여 에칭하여 제거함으로써, 내부가 비어있는 쉘 구조를 갖는 다이아몬드를 제조할 수 있는 방법을 제공하는 데 그

목적이 있다.

【발명의 구성】

이와 같은 목적을 달성하기 위하여, 본 발명은 두 단계에 걸쳐서 다이아몬드 쉘을 제조할 수 있다.

첫 번째 단계는, 크기가 수 ㎛~수 ㎜의 범위 내에 있는 실리카 모재 위에 수 ㎛ 두께의 다이아몬드 막을 CVD법으로 증착하는 것이다. 사용될 수 있는 다이아몬드 합성장치는 기존에 알려진, hot filament CVD, microwave plasma CVD 또는 direct current plasma CVD가 될 수 있으며, 합성 조건 역시 기존 다이아몬드 막합성에서의 것과 유사하게 유지될 수 있다. 합성초기 다이아몬드 핵생성 속도 및 핵밀도 증진을 위해, 다이아몬드 합성 전에 기존 CVD 합성법에서 널이 행해지는 전처리 (미분의 다이아몬드가 분산된 알콜이 담겨진 비커에 모재를 담구어, ultra sonic bath에서 일정 시간동안 진동시켜 모재의 표면에 scratch를 형성시켜 핵생성을 촉진시키는 방법) 또는 기판에 바이어스를 가할 수 있다.

다이아몬드 막 합성시, 모재의 일부분은 다이아몬드 막이 증착되지 않아야 하는데, 이 부분은 CVD법 다이아몬드 합성에서 모재가 놓여지는 판과 접촉하고 있는 실리카 입자의 아래면(기상과 접촉되지 않는 부분)이 자연스럽게 될 수 있다(도 2 참조). 또한 핵생성 속도 및 핵밀도 증진을 위한 미분의 다이아몬드입자를 사용하는 전처리 과정에서 실리카 모재의 일정부위를 미분의 다이아몬드 입자가 닿지 않도록 하여 CVD 다이아몬드입자가 핵생성하지 못하도록 하는 방법을 쓸 수 있다.

두 번째 단계는 다이아몬드 막이 증착된 실리카를 HF 수용액에 담가, 실리카를 에칭 하여 제거하는 것이다. 이 과정에서 에칭속도를 증가시키기 위해 HF 수용액을 가열하거나, 초음파 진동을 부과할 수 있다.

이러한 두 단계를 통하여, 내부가 비어 있는 다이아몬드 쉘을 제조할 수 있다. 또한, 다이아몬드 합성단계에서, 모재의 모양을 구, 사면체, 육면체 등으로 변화시키고 각 모재의 크기를 변화시킬 경우, 크기 및 구조가 다양한 다이아몬드 쉘을 제조할 수 있다. 또한 다이아몬드 합성조건을 변화시켜 다이아몬드 막의 표면 조직

을 제어함으로써, 다양한 조직 및 이에 따른 다양한 표면특성을 갖는 다이아몬드 쉘을 제조할 수 있다.

이하에서는 첨부된 도면을 참조하여 본 발명을 상세히 설명한다.

도 1은 본 발명의 단계를 나타낸 것으로, 구상의 실리카(1) 표면에 CVD법으로 다이아몬드 막(2)을 합성한다(가, 나). 다이아몬드 막 합성 후 실리카(1)를 HF 수용액에 담가 에칭 하여 제거함으로써, 내부가 공동(3)인 구상인 다이아몬드 쉘(4)을 얻을 수 있다(다).

도 2는 기상합성에 의한 다이아몬드 합성장치의 예를 나타낸 것인데, 이것은 일반적인 CVD 다이아몬드합성장치와 동일하다. 진공용기(5) 내에서 가스(수소+메탄)를 활성화시키기 위해 플라즈마(6)가 형성되며, 모재로 사용될 실리카(1)는 판(7) 위에 놓여져 있다. 다이아몬드합성의 주요 합성 변수인 가스 조성, 압력 및 증착온도는 각각 수소 가스내 1~20% CH_4, 10~200 Torr 및 약 600~1000℃로 제어된다. 다이아몬드는 모재에 핵생성하여 입자형태(8)로 생성하며, 합성이 경과됨에 따라 점차 도 1에 나타낸 바와 같이 다이아몬드 막(2)으로 성장한다. 막 두께는 합성시간 조절에 의해 조절될 수 있다. 이 그림에서 알 수 있는 바와 같이, 막 형성 단계 이전에 다이아몬드 합성을 중단할 경우, 합성시간에 따라 나노에서 마이크론 단위를 갖는 입자형태의 CVD 다이아몬드도 얻을 수 있다. 또한, 모재의 온도, 가스조성을 변화시켜, 형성되는 다이아몬드의 입자 및 막의 조직을 제어할 수 있다.

도 2에 나타낸 것은 플라즈마를 이용하는 합성장치의 예를 나타낸 것인데, 플라즈마는 직류전원, 마이크로웨이브 등에 의해 형성될 수 있다. 이들 장치는 기존 CVD법에 잘 알려져 있다. 또한, 플라즈마가 형성되지 않는 다이아몬드 합성장치인 hot filament CVD장치도 사용될 수 있다.

이하에서는 구체적인 실시 예를 들어 본 발명을 보다 상세히 설명할 것이나, 본 발명의 범위가 하기의 실시 예에 의하여 한정되는 것은 아니다.

< 실시예 1 >

마이크로웨이브 플라즈마 CVD법을 이용하여, 직경 50 ㎛인 실리카 구(silica

sphere) 위에, 10 시간 동안 다이아몬드를 합성하였다. 합성조건은 메탄조성 1%, 압력 40 Torr, 기판온도 650 ℃, 가스유량 200 sccm이었다. 주사전자현미경(SEM)을 이용하여 실리카 표면을 관찰한 결과 다이아몬드 막이 실리카 표면의 대부분을 덮고 있었으나, 일부는 연속적인 다이아몬드 막이 형성되지 않은 채 다이아몬드 섬(island)들이 관찰되었다. 다이아몬드 막의 표면 조직은, 일부는 (100)면이 주로 형성된 조직, 일부는 (100)면과 (111)면이 혼재된 조직, 그리고 일부는 특정 면이 형성되지 않은 nano-structured 조직을 보였다. 이러한 샘플에 따른 조직변화는 합성시 증착온도 및 플라즈마 intensity가 위치에 따라 일정하지 않았음을 나타낸다. 샘플을 HF 수용액에서 24시간 동안 에칭하였다. 에칭 온도는 60 ℃이었다. 에칭 후 주사전자현미경을 이용하여 샘플을 관찰한 결과 내부가 비어있는 다이아몬드 쉘을 관찰할 수 있었다. 쉘 벽의 두께는 5~10 ㎛이었다. 또한 일부의 다이아몬드 입자를 관찰할 수 있었는데, 이는 연속적인 다이아몬드 막을 이루지 못한 샘플(다이아몬드 섬)에서 얻어진 것이다.

< 실시예 2 >

실시예 1과 같은 조건으로 8 시간 동안 다이아몬드를 합성하였다. 주사전자현미경을 이용하여 실리카 표면을 관찰한 결과 다이아몬드 막이 부분적으로 관찰되었다. 이 샘플을 HF 수용액에서 24시간 동안 에칭하였다. 에칭 온도는 60 ℃이었다. 에칭 후 주사전자현미경을 이용하여 샘플을 관찰한 결과 다이아몬드 입자들이 관찰되었다. 다이아몬드 입자의 두께는 4~8 ㎛ 이었다.

【발명의 효과】

본 발명은 종래의 기술로는 제조할 수 없었던 내부가 비어 있는 다이아몬드 쉘(shell)을 제조할 수 있는 방법을 제공할 수 있다. 이러한 다이아몬드 쉘은, 다이아몬드의 고유특성인 화학적으로 안정함과 biocompatible 특성과 함께, DNA나 박테리아와 같은 바이오물질을 보존하는 용기나, 로숀이나 크림 형태의 약품을 담아 시간에 따라 일정하게 공급할 수 있는 용기로 사용될 수 있다. 또한, DNA 분리 및 검출기, field emission display(FED)의 전극재료, 등에 응용될 수 있다.

【특허청구범위】

【청구항 1】

CVD 다이아몬드합성법에서, 기하학적인 형상을 갖는 모재의 표면에, 표면의 일부를 제외하고, 다이아몬드 막을 증착한 후, 모재를 에칭하여 제거함으로써 내부가 비어있는 다이아몬드 쉘을 제조하는 방법.

【청구항 2】

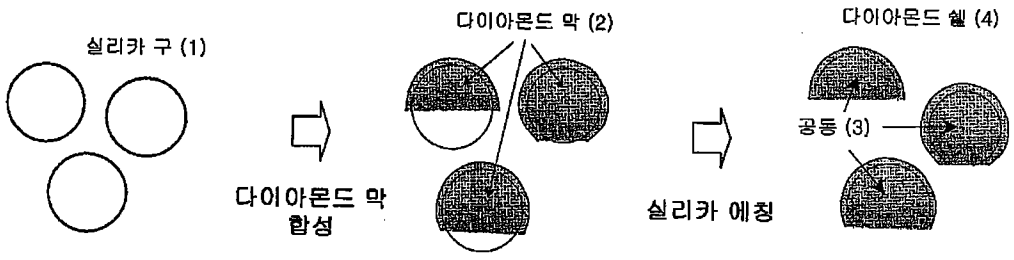
제 1 항에 있어서, 모재는 CVD 다이아몬드 합성이 가능하며, 화학약품에 의해 에칭 되는 재료이며, 모재의 모양은 구형, 사면체, 육면체 등으로 다양하게 변화될 수 있다.

【청구항 3】

제 1, 2 항에 있어서, 모재의 크기는 수 ㎛ ~ 수 mm이다.

【도면】

【도 1】



(가) (나) (다)

【도 2】

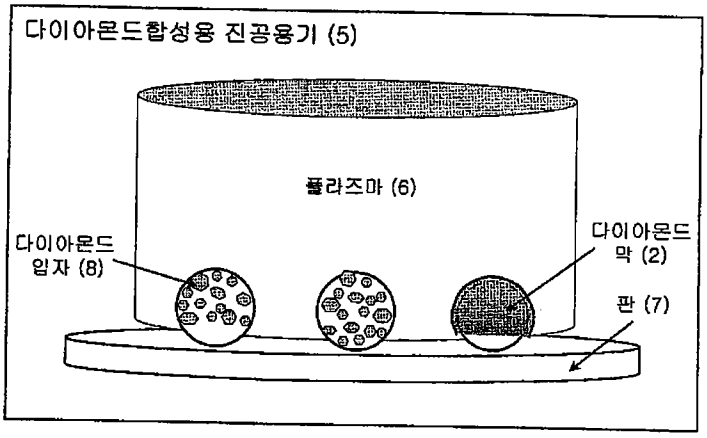


EXHIBIT 6

【ABSTRACT】

A method for fabricating micro-sized diamond shells is disclosed. Diamond film is partially deposited on micro-sized silica (matrix) by employing chemical vapor deposition (CVD) method and the silica is etched out for removal, to thereby obtain diamond shells with hollow inner space. Diamond shells with various shapes and sizes can be fabricated by varying the shape of matrix into sphere, tetrahedron and hexahedron or varying the size of matrix. The size of diamond shells to be fabricated is in the range of several ㎛ and several ㎜.

【Representative Drawing】

Figure 1

【Index】

Diamond, CVD, micro, shell, etching

【DESCRIPTION】

【Invention Title】

A METHOD FOR FABRICATION OF DIAMOND SHELLS

【Description of Drawings】

FIG. 1 shows a schematic view of the process making diamond shells by partially depositing, by using chemical vapor deposition method, diamond film on spherical silica 1 as a matrix, and etching the silica 1 out for removal, in accordance with one example of the present invention; and

FIG. 2 shows a schematic view of a diamond deposition on spherical silica by employing typical CVD diamond deposition apparatus.

**** Explanation for the major reference numerals ****

1: matrix (silica sphere)	2: diamond film
3: opening	4: diamond shell
5: diamond deposition chamber	6: plasma
7: plate	8: diamond particle

【Technical Field】

The present invention relates to a material field (ceramics), and more particularly, a method for fabricating hollow diamond shells by applying CVD method to diamond as one of ceramics.

【Background Art】

CVD diamond deposition method of which condition is about 100 Torr of pressure and 800°C of temperature was developed in the ^{1980th}. The CVD method

now becomes a technique typical enough to fabricate various CVD diamonds applicable to industries. By employing the CVD method, a source gas (usually, methane) is activated by being boiled or by plasma in a vacuum chamber, diamond nucleates on a matrix in a particle type, and grows to be polycrystalline films. Types of CVD methods may be classified according to how to activate a source gas. Several deposition methods have been developed and used so far, including a hot filament CVD, a direct current plasma CVD, a microwave plasma CVD, a direct current arc jet CVD and the like. Typically, diamond film is deposited on a plate-like matrix (substrate) with several ㎚ to hundreds ㎛ in thickness. Such diamond film is used with being attached onto the matrix or used as a freestanding diamond film independent from the matrix. As such, a diamond film is generally obtained by the CVD method.

High pressure and high temperature (HPHT) method, of which condition is 1400℃ of temperature and about 50,000 atmosphere of pressure, developed by GE Ltd., in the United States in 1955 prior to the CVD method, enables us to synthesize grit-type diamonds in a shape of cubo-octahedron whose sizes are below hundreds ㎛ in diameter or irregular shaped diamond powders. Diamond obtained by employing a single crystal growth method carried out under the HPHT conditions is a bulk type with several ㎟ in size. Also, detonation method developed in Russia of which condition is 1,000℃ of temperature and about 100,000 atmosphere of pressure enables us to synthesize irregular diamond powders with several ㎚ to hundreds ㎛ in size. So, diamond powders which are similar to natural diamonds are typically obtained by adopting the HPHT method. However, since diamond is a high pressure phase of carbon, the deposition conditions of the HPHT method are severe, resulting in limitation of shapes of

diamonds to be obtained under such conditions.

CVD method depositing diamond films below atmospheric pressure brought an opportunity for much overcoming the limitation of sizes and shapes of diamonds, which allowed fabricating of diamonds with diameters in the range of several to tens ㎠, or dome-shaped freestanding diamonds with diameters of several ㎠ (and also with thickness below about 2 ㎜).

However, the existing CVD deposition technique could not allow fabricating of diamond shells with hollow inner space with micrometers in size.

【Disclosure】

【Technical Solution】

Therefore, the present invention is proposed to fabricate diamond shells which cannot be fabricated by the related art technique, in particular, to provide a method for fabricating diamond shells with desired sizes in the range of several ㎛ to several ㎜. An object of the present invention is to provide a method for fabricating hollow diamond shells by partially depositing diamond film with preset thickness on a spherical matrix (silica) in the range of several ㎛ to several ㎜, and etching the partially deposited matrix using chemicals.

【Mode for the Invention】

To achieve this advantage of the present invention, the present invention proposes two processes of fabricating diamond shells.

The first process is to employ CVD method to deposit diamond films with a thickness of several ㎛ on a silica matrix in the range of several ㎛ to several ㎜ in size. An apparatus to be used for synthesizing a diamond film is such as a hot

filament CVD apparatus, a microwave plasma CVD apparatus, or a direct current (DC) plasma CVD apparatus. Similar synthesizing conditions to the conventional methods can be applied. In order to promote nucleation of diamond and nuclear density at the initial stage of synthesis (deposition), diamond may be pre-treated before synthesis or a matrix may be biased. Here, the pre-treatment denotes a method in which a matrix is put into a beaker containing alcohol with find diamonds being dispersed therein, and vibrated in an ultrasonic bath for a preset time to scratch the surface of the matrix, thereby promoting nucleation.

Upon synthesizing (depositing) diamond films, the matrix must partially be uncovered with diamond films. The partially uncovered surface may be used as a bottom zone (a surface not in contact with a gas phase) of a silica particle, which is in contact with a plate on which a matrix is placed upon the diamond deposition using CVD method (see FIG. 2). Also, during the pre-treatment using the fine diamond particles for promoting nucleation speed and nuclear density, a partial surface of silica matrix is blocked from fine diamond particles, thereby preventing nucleation of CVD diamond particles.

The second process is to put a diamond film deposited silica in HF solution and etch the silica out for removal. During this process, in order to increase etching speed, the HF solution may be boiled or ultrasonic vibration may be applied.

Through the two processes, hollow diamond shells can be fabricated. Further, during diamond synthesis, diamond shells with various sizes and structures can be fabricated by varying the shape of matrix into sphere, tetrahedron, hexahedron and the like and varying the size of each matrix. Also, a surface morphology of diamond films can be controlled by varying diamond

synthesis conditions, thereby diamond shells being fabricated with various morphologies and surface characteristics.

Hereinafter, the present invention will be described in detail with reference to the accompanying drawings.

FIG. 1 shows the processes of the present invention. Diamond film 2 is deposited on surfaces of spherical silica 1 by employing CVD method (A and B). After the deposition of the diamond film, the silica 1 is put into HF solution to etch the silica 1 out for removal, thereby spherical diamond shells 4 with hollow inner opening 3 being obtained (C).

FIG. 2 shows an exemplary diamond deposition apparatus by synthesis of a gas phase. This apparatus is the same as a typical CVD diamond deposition apparatus. Plasma 6 is generated in a vacuum chamber 5 to activate a gas (i.e., H2 + methane). Silica 1, as a matrix, is placed on a plate 7. Main conditions for the diamond deposition are controlled as follows: 1~20% CH_4 in the source gas, 10~200 Torr of pressure, and about 600~1000℃ of deposition temperature. The diamond is nucleated on the matrix and thereby diamond particles 8 are generated. During deposition, the diamond particles 8 are gradually grown to be diamond film 2 as shown in FIG. 1. The thickness of films may be controlled by varying the deposition time. As shown in the drawings, if we stop the diamond deposition before the film formation, CVD diamond particles can also be obtained with nanometers to micrometers in size depending on the deposition time. Also, matrix temperature and gas composition can be varied to control the structures of diamond particles and films formed.

FIG. 2 exemplarily shows a deposition (synthesis) apparatus using plasma.

Plasma may be formed by direct current, microwaves and the like. Such apparatuses are well-known CVD apparatuses. Also, hot filament CVD apparatus which is a diamond deposition apparatus without plasma being formed may be employed.

Hereinafter, the present invention will be described in more detail with reference to the detailed examples; however, the scope of the present invention may not be limited to the following examples.

<Example 1>

Diamond film was deposited on silica spheres with 50 ㎛ in diameter by employing a microwave plasma enhanced CVD apparatus under the condition of 1 % methane composition, 40 Torr of pressure, 650℃ of substrate temperature and 200 sccm of flow rate for 10 hours. In SEM observation, some silica spheres revealed diamond films covered partially thereon. Others revealed diamond islands without diamond films being continuously covered thereon. Surface morphologies were (100) dominant, (100) and (111) facets mixed and partially nano-structured without a specific surface being formed. Such variation of morphologies depending on samples reveals that deposition temperature and plasma intensity during deposition are not fixed with respect to positions. The samples were etched out in HF solution at 60℃ of temperature for 24 hours. In SEM observation of the samples after being etched, diamond shells with hollow inner spaces were revealed. The thickness of shell walls was in the range of 5 ~ 10 ㎛. Diamond particles were also partially observed, which were obtained from the sample (i.e., diamond islands) without diamond films being covered thereon.

<Example 2>

A diamond deposition was performed for 8 hours under the same condition of Example 1. In the SEM observation, silica surface partially revealed diamond films. This sample was etched out in HF solution at 60℃ of temperature for 24 hours. In the SEM observation of the sample after being etched, diamond particles with thickness of 4 ~ 8 ㎛ were observed.

【Effect of the Invention】

The present invention can provide a method for fabricating diamond shells with hollow inner space which couldn't be fabricated in the related art. Such diamond shells can reveal chemical stability as a specific characteristic of diamond and biocompatibility. The diamond shells are useable as a container for preservation of biomaterials, e.g., DNA or bacteria, or as a container uniformly supplying chemicals in creams or lotions based on time. Also, the diamond shells can be applied to DNA separator and detector, electrode materials of field emission display (FED) and the like.

【CLAIMS】

1. A method for fabricating diamond shells with hollow inner space by partially depositing diamond film on a surface of a matrix with a geometrical shape by employing a chemical vapor deposition (CVD) diamond deposition method, and etching the matrix out for removal.

2. The method of claim 1, wherein the matrix supports CVD diamond deposition, and is formed of a material etched by chemicals, the shape of the matrix being variously changed into sphere, tetrahedron, hexahedron and other shapes.

3. The method of claims 1 and 2, wherein the size of the matrix is in the range between several ㎛ and several ㎜.

EXHIBIT 7

K03344

발명신고서(이재갑)

* 인적사항 정보 추가

발신부서	★-	-	센터장	부장		
	이재갑	최원국	교육	07/12 김준경		

수신부서	-	담당
	조선희	07/12 민경남

협조										

발명의 명칭	국 문	마이크로 다이아몬드 쉘의 제조 방법		
	영 문	A method for the fabrication of micro-diamond hollow shells		
출원경비	출원희망국	금 액	계정번호	
	한국	1000000	2E18160(일반) OK	
		나머지 1,000,000은 특허개발협의서 참조		
연구과제		연구기간		
		계정번호		
발명의 공표 (예정포함)	발표매체	발 표 제 목	발표(예정)일	
발명구분	직무발명			

이 발명은 직무발명으로서 신고하오며 위발생에 관하여 국내 및 국외에서 특허발을 수 있는 모든 권리는 양도함을 확약합니다.

특허사무소	,박장원

[발명신고서일 경우]

위와 같이 발명을 신고하오니 승계하여 주시기 바랍니다.

붙임 : 1. 명세서

2. 산기협 제출서류 각 1부
(KIST 소유과제일 경우)
- 기술설명서
- 기술개발개요
- 명세서
- 선행기술조사서
- 특허경비지원사업확약서

[프로그램등록신고서일 경우]

위와 같아 프로그램을 신고하오니 승계하여 주시기 바랍니다

붙임 : 1. 프로그램 등록 신청서

2. 프로그램 개요

3. 위임장

2004.07.08.

한국과학기술연구원장 귀하

다음 페이지로

EXHIBIT 8

STATEMENT OF INVENTION (LEE JAE KAP)

Title of invention	Korean	마이크로 다이아몬드 쉘의 제조 방법		
	English	A method for the fabrication of micro-diamond hollow shells		
Application Fee	Expected Country of Application		Payroll	Account No.
	Korea		1000000	2E18160(general)
Type of Invention	In-service Invention			
This invention is reported as an in-service invention and we confirm to assign every right for internal and external patent as to the invention stated above.				
Attorney	JANG WON, PARK			

[For statement of invention]

We would like you to take over the invention stated above.

Enclosure: 1. Specification

2. Each copy of documents submitted in Korea Industrial Technology Association
- Technical description
- Overview of technical development
- Specification
- Prior art examination
- Executed letter of support project for patent expense

[For statement of program registration]

We would like you to take over the program stated above.

Enclosure: 1. Application of program registration

2. Overview of program

3. Letter of Attorney

Date posted: July 8, 2004

President

Korea Advanced Institute of Science and Technology

EXHIBIT 9

발명 신고서 및 양도증 서명서

발명의 명칭	국 문	마이크로 다이아몬드 쉘의 제조 방법
	영 문	A method for the fabrication of micro-diamond hollow shells

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	직급		영 문 명	
	지분		주 소	
	소속		주민등록번호	
	직급		영 문 명	
	지분		주 소	
	소속		주민등록번호	
	직급		영 문 명	
	지분		주 소	
	소속		주민등록번호	
	직급		영 문 명	
	지분		주 소	
	소속		주민등록번호	
	직급		영 문 명	
	지분		주 소	
	소속		주민등록번호	
	직급		영 문 명	
	지분		주 소	
	소속		주민등록번호	
	직급		영 문 명	
	지분		주 소	
	소속		주민등록번호	
	직급		영 문 명	
	지분		주 소	

이전 페이지로

EXHIBIT 10

STATEMENT OF INVENTION AND ASSIGNMENT

Title of the Invention	Korean	마이크로 다이아몬드 쉘의 제조 방법
	English	A method for the fabrication of micro-diamond hollow shells

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